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Papermaking characteristics of three *Populus* clones grown in the north-central United States

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Abstract

The papermaking properties of 22 pure and hybrid poplars are being evaluated in an on-going investigation. Twenty of the poplars were harvested after 7.5 years from three different sites in the Midwestern and North Central US. The other 2 poplars survived at only two of three sites (64 total samples). The Crandon hybrid had the highest growth rate ($t\,ha^{-1}\,y^{-1}$) and wood density (both averaged across the 3 sites). This poplar had a high cellulose content (compared to the average), a low lignin content and produced bleached kraft fibers at a high yield (wt.% on wood chips). Further, this poplar responded very well to kraft pulping and oxygen delignification and bleached to the highest final brightness ever observed in our laboratory (94.5% Elrepho). It also produced an 18 kappa number unbleached pulp with $<0.5\%$ rejects in only two-thirds the time required for sugar maple (*Acer saccharum*).

We also report on clone 220-5 that had the highest area-weighted average microfibril angle. Pulps from this poplar had excellent tensile properties and further improvements are expected with 1–2 years of additional growth that should result in a small, but significant increase in average fiber length. Some results are also presented for clone 313.55 and aspen (*Populus tremuloides*) to demonstrate the many substantial benefits that can be accrued from proper wood selection.

Keywords: Kraft pulps; *Populus*; Lignin; Microfibril angle; Fiber properties

1. Introduction

According to some projections there may be regional shortages of hardwood papermaking fibers starting around 2010 [1,2]. There are many publications showing that several fast-growing hybrid poplars do produce high-quality pulps from the dominant kraft process. However, their low wood density is a major drawback to commercial use. The capital cost associated with kraft digester capacity (volume) is quite high and pulp is sold by weight. Therefore, for a given digester volume sugar maple chips with basic density (oven-dried wt./water swollen volume) of $\sim 550\,kg\,m^{-3}$ [3] would produce ~60% more pulp by weight as compared to a hybrid poplar with basic density of $\sim 350\,kg\,m^{-3}$. For a hybrid poplar to be used in the kraft process it would have to possess other superior attributes in addition to high growth rate when compared to the traditional North American hardwoods. One such attribute that appears to be quite common in hybrid poplars (<15 years old) is a high-tensile energy absorption (TEA) during papermaking [4]. The TEA (at the point of rupture) for kraft sheets from an 11 year-old DN 30 (*Populus deltoides* × *P. nigra*) was 75% higher than for sheets from mature aspen (*P. tremuloides*) [4]. There are enormous tensile stresses on modern paper machines running at $30–50\,m\,s^{-1}$. Therefore, a 75% increase in TEA would represent a significant increase in strength and should allow for less fiber and more inexpensive fillers ($CaCO_3$) to be used in papermaking.

Twenty-two poplars grown on three different sites are being evaluated for their wood and papermaking

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properties in a three-year investigation. The three sites are Arlington, WI (ARL), Iowa State University, Ames, Iowa (ISU) and Westport, MN (WPT). Data on parentage and growth rate for all the poplars are documented by Riemenschneider et al. [5]. A preliminary report is now being given and it is focused on pulps from Crandon, 220-5, and 313.55 clones. The Crandon clone had the highest growth rate [5] and basic wood density ($399\,kg\,m^{-3}$ versus an average of $330\,kg\,m^{-3}$) while the 220-5 clone had the highest area-weighted MFA (30.9° averaged across 3 sites) and above average basic density ($353\,kg\,m^{-3}$). The 313.55 clone along with aspen was included for additional data so that possible trends would be more clear.

2. Materials and methods

2.1. Wood supply and pulping

The Crandon hybrids *(P. alba × P. grandidentata)*, 313.55 hybrids (*P. deltoides × P. maximowiczii*) and 220-5 *deltoides* clones were provided by Dr. Riemenschneider [5]. Aspen (31 years old) and mature sugar maple were harvested on properties owned by SUNY-ESF in Central New York. The logs were debarked and converted to chips. Kraft pulping is used to extract 90–95% of the lignin from the chips and was conducted in pressurized digesters with liquor circulation. A 5:1 cooking liquor to wood ratio was used with 16% active alkali on chips (NaOH and Na_2S added on a Na_2O basis) and 25% sulfidity, i.e. Na_2S/(NaOH + Na_2S). For most of the cooks, the time to reach the maximum temperature of 165 °C was 90 min and the duration at 165 °C was 120 min. For the rapid impregnation-short time (RIST) pulping study, the cooking liquor was similar to that above but the time to and time at 170 °C was varied.

2.2. Bleaching by OD_0EpD_1 sequence

Oxygen: Conducted in a Quantum Mark IV reactor at 12% consistency, 0.72 MPa of O_2, 2.0% NaOH, and 0.5% $MgSO_4 \cdot 7H_2O$ on pulp at 90 °C for 1 h. An O-stage is typically used to remove 50% of the residual lignin in the fibers.

D_0 stage: In plastic bottles at 3.0–3.5% consistency, 70 °C, 2 h with initial pH ~3.5 (before the addition of $C1O_2$) and end pH 2.5–3.0. A kappa factor of 0.20 (% equiv. $C1_2$/incoming kappa number) was used on all occasions.

Ep stage: In plastic bags at 12% consistency, 80 °C, 2 h with 2.0% NaOH, 0.25% H_2O_2 and 0.1% $MgSO_4 \cdot 7H_2O$ on pulp. The end pH was always in the range of 11.2–11.6. The lignin content of the pulp is typically <0.5 wt% after this stage.

D_1 stage: In plastic bags at 10% consistency, 70 °C, 3 h, 0.4% $C1O_2$ on pulp and a small amount of NaOH or H_2SO_4. The end pH was always in the range of 3.5–4.1.

2.3. Analyses by Tappi standard methods

The following analyses were by Tappi standard methods: Wood density by water displacement method, ethanol/benzene extraction, lignin content (Klason and acid soluble), kappa number (estimation of lignin content), viscosity (estimation of cellulose DP), and pulp strength.

2.4. Other analyses

Microfibril angle was determined by X-ray diffraction and wood density by X-ray densitometry using SilviScan-2. These analyses were performed by CSIRO Forestry and Forest Products, Tasmania, Australia [6,7].

Carbohydrate composition analysis was performed by the Analytical and Technical Services (ATS) group at SUNY-ESF using a ^1H NMR method they developed [8]. The method involves treating 40 mg of extractive-free woodmeal with 0.4 mL of 72% H_2SO_4. After stirring, the dispersion was allowed to digest at 40 °C for 1 h in a water bath or oven with additional stirring every 15 min. Following the digestion, 2.0 mL of D_2O (NMR solvent) was added to the dispersion, which was then autoclaved in a high-pressure sealed glass tube at 121 °C for an additional 1 h. Hydrolyzates prepared in this fashion were then filtered into NMR tubes without neutralization of the H_2SO_4. Since the H_2SO_4 employed in the analysis contains approximately 28% H_2O, the samples tested by NMR contained approximately 5% water (in D_2O) which could potentially interfere with the observation of ^1H signals resulting from the sugars. The low pH of the acidic hydrolysis medium shifted the "water" NMR peak (due to HDO) away from the region of C-1 anomeric protons, which were used to quantify the biomass-derived sugars. Specific NMR details and conditions include: Bruker AVANCE 600 MHz NMR system (proton frequency = 600.13 MHz), Broadband observe probe (BBO), 30 °C, 90° pulse = 11 μs, recycle time = 10 s, acquisition time = 2.73 s, sweep width = 10 ppm, center of spectrum = 4.5 ppm, reference = acetone at 2.2 ppm (1 μL ladded to sample tube). No peaks are detected for acetyl and uronic acid/anhydride groups. A constant total value of 7.0 wt% based on unextracted wood [9] was assumed for all the poplars.

Brightness was determined on an Elrepho brightness meter while a Kajaani FS 100 analyzer was used to determine fiber coarseness and weight (mass)-weighted average fiber length (AFL), i.e. $L_w = (\sum n_i L_i^3)/(\sum n_i L_i^2)$, where $i = 1, 2, 3, \ldots, n$ is the fiber count and L the contour length.

3. Results and discussion

The chemical composition data are presented in Table 1. Woodmeal (40 mesh) was first extracted with ethanol/benzene and the extractives content quantified. The extracted woodmeal was then analyzed for lignin and

Table 1
Chemical composition of woodmeals[a]

Sample	Extractives	Lignin	Cellulose	Xylose	Other hemicelluloses[b]
Crandon (ARL)	2.4	18.1	48.6	18.7	12.2
Crandon (ISU)	2.2	18.6	48.5	20.1	10.6
Crandon (WPT)	1.5	19.0	47.9	20.7	10.9
220-5 (ARL)	0.9	19.6	49.0	18.1	12.4
220-5 (ISU)	0.7	21.6	44.1	20.3	13.3
220-5 (WPT)	1.5	22.8	43.8	19.4	12.5
313.55 (ARL)	1.1	21.9	48.5	17.1	11.4
313.55 (ISU)	1.3	22.8	46.8	17.7	11.4
313.55 (WPT)	1.9	22.7	46.4	17.2	11.8
Aspen	2.3	18.6	49.6	18.4	11.1

[a]wt% based on unextracted woodmeal.
[b]Sum of mannose, galactose, arabinose, acetyl and uronic acids.

Table 2
Physical properties of solid woods

Sample	Basic wood density, $\mathrm{Kg\,m^{-3}}$		MFA, deg
	Tappi[a]	X-ray[b]	
Crandon (ARL)	400	399	19.7
Crandon (ISU)	379	371	17.1
Crandon (WPT)	415	429	19.3
220-5 (ARL)	351	350	30.7
220-5 (ISU)	339	347	29.8
220-5 (WPT)	357	373	32.1
313.55 (ARL)	290	264	21.0
313.55 (ISU)	312	314	18.4
313.55 (WPT)	319	305	—
Aspen 1[c]	—	363	14.7
Aspen 2	—	369	12.5

[a]Determined in Syracuse (see Section 2).
[b]Determined in Australia (see Section 2).
[c]Two 31-year-old trees from the same plot of land.

carbohydrates. It should be noted that the precision on lignin content was about $\pm 0.5\%$ on woodmeal based on two to five analyses per woodmeal sample.

Two noteworthy observations were the consistently high cellulose contents for the Crandon clones and the higher cellulose content for 220-5 grown at Arlington as compared to the same clone planted at the other two sites. A significantly higher cellulose content ($>2\%$ on wood) for the Arlington site was observed for ~10 of the 22 poplars.

The MFA and basic density measured on solid wood bolts are presented in Table 2. The Crandon and 313.55 hybrids had low MFA's while the values were significantly higher for the 220-5 clones. Other important fiber properties such as length and coarseness ($\mathrm{mg\,m^{-1}}$) were determined using kraft pulp fibers and will be presented and discussed later. The MFA is the average angle of the cellulose microfibrillar helix in the S2 layer of the cell wall, relative to the fiber axis [6]. There are credible data suggesting that when a tensile load is applied to sheets from refined hardwood kraft pulps the % strain (to the point of rupture) increases with MFA [4,7,10]. This increased strain occurs at a high stress level [4] and results in a significantly higher TEA (obtained from the area under the stress–strain curve).

3.1. *Kraft pulping and bleaching*

Kappa number (wt% lignin $\approx 0.15 \times$ kappa number) and screened yield for the unbleached pulps are presented in Table 3. All three Crandon pulps afforded a screened fiber yield $>55.0\%$ while the average value (across 3 sites) was 54.7% for the 220-5 and 54.9% for the 313.55 pulps with a significantly higher value for the ARL site as compared to WPT. There was an expected correlation between cellulose content and pulp yield as shown in Fig. 1. Data from another clone (across all 3 sites) were included to extend the range of the correlation. The Crandon pulps also achieved lower kappa numbers than the 220-5 and 313.55 clones.

Aspen is probably the most widely used poplar for papermaking in North America. It produced a pulp with kappa number of 16.7 at 57.1% yield. Its response to kraft pulping was slightly inferior or equal to the Crandon clones but slightly superior to the 220-5 and 313.55 clones.

The pulps were then delignified with oxygen and the Crandon clones were once again quite responsive to the treatment. The bleaching results are presented in Table 4 and it can be seen that the post O_2 kappa numbers of the Crandons were significantly lower (>1.0 kappa unit) than for all of the other pulps. The importance of post O_2 kappa number is demonstrated in Fig. 2 where it is plotted against final brightness. The oxidizing equivalents from chlorine dioxide that were applied in the D_0 stage were proportional to the kappa number after O_2 (See Section 2). Therefore, a pulp with a lower post O_2 kappa number required less ClO_2, but gave a higher final brightness (Fig. 2). It appears that the nature of the Crandon lignin is more amenable to pulping and bleaching reactions. A higher syringyl to guaiacyl ratio is suspected for the Crandon clone because syringyl units are known to depolymerize at a significantly

Table 3
Kraft pulping results

Sample	Kappa number	Screened yield[a]
Crandon (ARL)	13.7	56.4
Crandon (ISU)	15.3	55.9
Crandon (WPT)	15.5	55.3
220-5 (ARL)	17.8	56.4
220-5 (ISU)	17.5	54.2
220-5 (WPT)	18.2	53.6
313.55 (ARL)	17.4	55.3
313.55 (ISU)	18.4	55.2
313.55 (WPT)	18.3	54.2
Aspen	16.7	57.1

[a]% on chips.

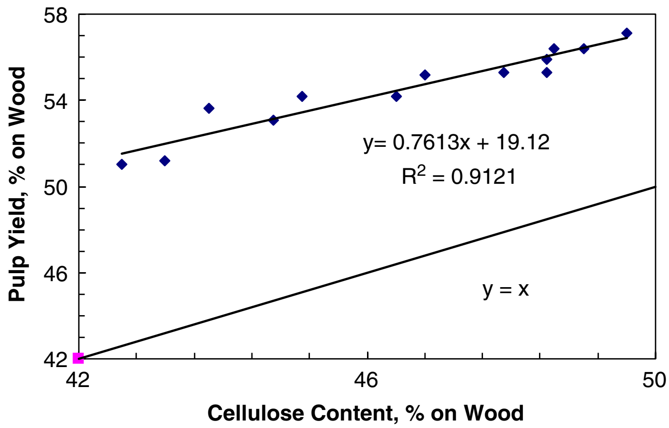

Fig. 1. Kraft pulp yield versus estimated cellulose content of wood chips from 13 poplars.

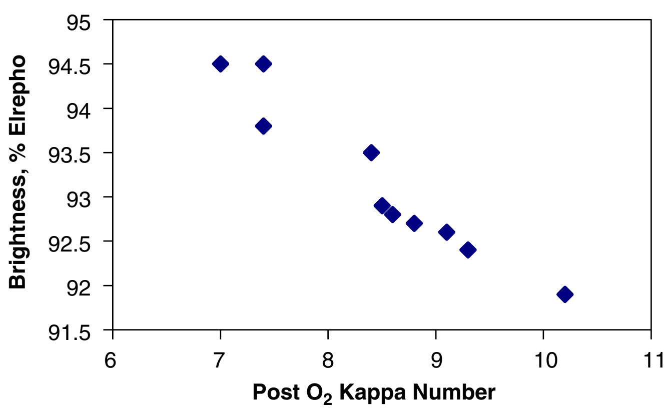

Fig. 2. Final brightness (after OD_0EpD_1) versus post O_2 kappa number for 10 poplar pulps with standard kraft pulping and oxygen delignification conditions.

higher rate during kraft pulping [11]. Syringyl groups remaining in the pulp would be oxidized at a higher rate during the O_2 stage due to the added methoxyl group that is electron-donating [12]. Finally all of the bleached pulps had a viscosity >18 cP (Table 4) and this normally indicates a relatively high degree of polymerization for the cellulose.

The significant variation in delignification rate amongst the poplars was somewhat unexpected and led to an investigation of shorter pulping times for the Crandon. The lower density of the poplars indicates that they are more porous than species with higher density. Diffusion of the pulping chemicals into the Crandon chips should be more rapid than for sugar maple chips. The chip size distribution was equivalent for all the hardwoods used in this investigation. If the Crandon also requires less time once the pulping temperature is attained then its overall retention may be much less than for sugar maple. The Crandon chips from ARL are compared to sugar maple chips in Table 5 using rapid impregnation—short time or RIST Kraft pulping. The digester capacity required to handle 550 kg of maple chips would handle 399 kg of Crandon chips. Based on the results in Table 5, the maple chips would produce 289.3 kg of pulp after 180 min (550×0.526) while 225.4 kg of Crandon pulp would be produced after 120 min (338 kg in 180 min). If a continuous digester were to be used then the production rate for the Crandon would be higher than for sugar maple. Continuous digesters perform more smoothly with chips of higher density because of the greater downward force of the chip column [13]. However, there are some mills that have continuous digesters and operate with 100% aspen for some customers. The wood density data are given in Table 2 and the values for the Crandon clones are higher than for the typical aspen.

If batch digesters were to be used then a constant time of ~40 min would have to be added to the pulping time. This would be for emptying and reloading of the digesters plus chip pre-steaming. In this case 289.3 kg of maple pulp would be produced in 220 min and 225.4 kg of Crandon in 160 min (310 kg in 220 min).

3.2. Physical properties of fibers and sheets

Fiber and strength properties are documented in Table 6. The AFLs for the young poplars (7.5 years old) were much

Table 4
Bleaching results for kraft pulps[a]

Sample	O_2 Kappa number	% O_2 delignification[b]	Final[c] brightness	Final[c] viscosity, cP
Crandon (ARL)	7.0	49	94.5	22.4
Crandon (ISU)	7.4	52	94.5	27.9
Crandon (WPT)	7.4	52	93.8	24.3
220-5 (ARL)	9.3	48	92.4	21.8
220-5 (ISU)	8.6	51	92.8	22.7
220-5 (WPT)	10.2	44	91.9	20.5
313.55 (ARL)	9.1	48	92.6	23.0
313.55 (ISU)	8.4	54	93.5	21.6
313.55 (WPT)	8.5	54	92.9	22.1
Aspen	8.8	47	92.7	—

[a]Bleached fiber yield of 95.5–96.0% on unbleached pulp in all cases.
[b]Based on kappa number reduction.
[c]After OD_0EpD_1.

Table 5
Kraft pulping at 170 °C with short retention times

Species	Total time, min	Kappa number	Screened yield	% Rejects
Sugar Maple	120 (60[a])	25.1	53.0	1.5
	160 (90)	22.5	53.3	0.6
	180 (90)	17.9	52.6	0.2
Crandon (ARL)	105 (45)	19.8	56.5	0.9
	120 (60)	16.5	56.3	0.4

[a]Time to maximum temperature of 170 °C.

Table 6
Fiber and strength properties of refined kraft pulps (unbleached pulps with one exception)[a]

Sample	CSF, mL	Sheet density, $g\,cm^{-3}$	Fiber length, mm	Coarseness, $mg\,m^{-1}$	Tensile index, $N\,m\,g^{-1}$	% Strain	TEA, $J\,m^{-2}$	Tear index, $mN\,m^2\,g^{-1}$
Crandon (ARL)	515	0.81	0.78	0.11	92	4.3	168	8.1
170 °C RIST Pulp	504	0.80	—	—	96	4.2	169	8.0
Crandon (ISU)	550	0.82	0.75	0.11	89	4.5	171	8.6
Crandon (WPT)	512	0.83	0.73	0.11	85	4.2	152	7.4
220-5 (ARL)	505	0.86	0.76	0.12	91	4.8	196	7.7
220-5 (ISU)[b]	424	0.83	0.80	0.12	90	5.1	202	9.6
220-5 (ISU)-O_2[c]	362	0.81	—	—	81	7.2	280	8.4
220-5 (WPT)	449	0.89	0.66	0.12	80	4.7	176	7.5
313.55 (ARL)	432	0.87	0.65	0.13	90	3.8	148	6.3
313.55 (ISU)	450	0.88	0.67	0.12	93	4.0	170	7.2
313.55 (WPT)	472	0.89	0.65	0.13	90	4.3	174	7.3
Aspen	—	0.82	0.98	0.13	93	3.4	132	7.6

[a]1500 PF1 revolutions at light load in most cases.
[b]Average of 3 analyses of 5 sheets each.
[c]Oxygen delignified, only 500 PF1 revolutions.

lower than for the 31-year-old aspen. However, fiber length is expected to increase with age and the research program calls for re-evaluation of these poplars after 8.5 and 9.5 years of growth. Tear index generally increases with AFL [10,14] and is the key strength parameter for hardwood kraft pulps. Tensile index continuously increases with refining. However, there is usually a maximum in tear index and this is usually observed at a tensile index of 90–100 $N\,m\,g^{-1}$.

The results obtained for aspen are very comparable to other published results. The maximum tear for aspen kraft is normally in the range of 7.5–8.0 $mN\,m^2\,g^{-1}$ [10,15]. Also, a % strain (at sheet rupture) of 3.0–3.4% is typical [10,15]. Despite their much shorter fibers most of the young Crandon and 220-5 poplars, except for the Westport site, had equal or higher tear strength than the aspen pulp. The 313.55 hybrids from all 3 sites had AFLs <0.70 mm and lower tear indices than the aspen.

The higher temperature used for the RIST process had no negative effect on the strength of the Crandon ARL pulp and this was the expected outcome. Pulps from ISU had higher tear indices and superior overall strength properties as compared to the other two sites. This was observed for ~15 of the 22 poplars. The tear index for the 220-5 ISU was significantly higher than for all other pulps. We do not have an explanation at this time, but we have great confidence in the data. Three analyses of five sheets each were performed on this pulp and all the data were close to the average reported in Table 6.

There were no significant differences in fiber coarseness and its effect on pulp strength is not clear. The general trend in Table 6 is that tear index increased with fiber length, i.e. with the exception of aspen. If the three poplars for ARL are compared the Crandon had the highest AFL and the highest tear while the 313.55 hybrid had the lowest AFL and lowest tear. When multiple regressions were performed (Minitab) on the overall data, AFL was highly correlated with tear index ($p = 0.009$) and AFL explained ~33% of the variation in tear index.

The 313.55 hybrid is considered to be an inferior poplar because of its low wood density and AFL after 7.5 years of growth.

Strain to the point of rupture is reported to increase by ~40% when hardwood kraft pulps are bleached [10]. The 220-5 ISU pulp was lightly refined after O_2 delignification (500 PF1 revolutions) and high tensile and tear indices were not attained. However, the sheets strained an average of 7.2% before rupturing and the TEA was 280 $J\,m^{-2}$ (Table 6). The 7.2% strain is the highest we have ever observed for either a hardwood or softwood kraft pulp. For comparison, sugar maple sheets with a tensile index of 91 $Nm\,g^{-1}$ had a strain of 4.0% and a TEA of 156 $J\,m^{-2}$. The softwood, loblolly pine (*Pinus taeda*) had a strain of 5.0% and TEA of 164 $J\,m^{-2}$ at a tensile index of 79 $Nm\,g^{-1}$. Both pulps were O_2-delignified. The area-weighted average MFA for the 220-5 ISU poplar was 29.8° (Table 2) and its strain result is consistent with those of Gurnagul et al.[10] for bleached hardwood pulps. Those authors correlated % strain with average MFA for six mature Canadian hardwood species. There was a linear correlation ($R^2 \approx 0.90$) between average MFA and %strain. Their highest strain value was 5.9% at an average MFA of 24.2° [10].

While only one of the six species analyzed by Gurnagul et al. [10] had an average MFA > 20°. A majority of the poplars involved in this study (41 of 64) had area-weighted averages > 20°. It is well known that the MFA of the annual rings for hardwoods decreases with age [16–19]. If you have fast-growing hardwoods that can be harvested after 8–10 years then you have a high probability of achieving a high area-weighted MFA and the tensile benefits associated with it.

4. Conclusions

The papermaking properties of 22 pure and hybrid poplars are being evaluated in an ongoing investigation. This preliminary report focuses on three of the poplars. One of them (Crandon) had the highest growth rate ($\mathrm{t\,ha^{-1}\,y^{-1}}$) and basic wood density while another (220-5) had the highest area-weighted microfibril angle (averaged across 3 sites). The Crandon also had a high cellulose and a low lignin content and gave a high fiber yield (wt% on chips) after pulping and bleaching. It was easily delignified during kraft pulping and bleaching (by the OD_0EpD_1 sequence) and produced a strong pulp. It had the highest bleached brightness ever observed in our laboratory (94.5% Elrepho).

The 220-5 clone with the highest MFA afforded kraft paper sheets with high strain and tensile energy absorption (to the point of rupture). These properties were equal or superior to any other hardwood kraft pulp previously investigated in our laboratory. These properties would be expected to increase even further with 1 or 2 years of additional growth that should result in a small, but significant increase in average fiber length.

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